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TECHNICAL REPORT NO. 27

(Tertiary-butyl)cyclopentadienylindium(I), $\text{In}(\text{C}_5\text{H}_4\text{CMe}_3)$ -Synthesis,
Characterization and X-Ray Structural Study

by

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Summary

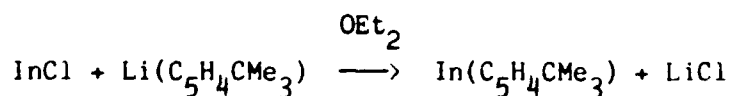
The indium(I) compound, $\text{In}(\text{C}_5\text{H}_4\text{CMe}_3)$, has been prepared from the corresponding lithium cyclopentadienyl reagent $\text{Li}(\text{C}_5\text{H}_4\text{CMe}_3)$ and InCl in OEt_2 . Characterization data have included partial elemental analyses (C, H), physical properties, IR, ^1H NMR and mass spectroscopic data, oxidation reactions with dilute aqueous HCl and a single-crystal X-ray structural study. The colorless crystal of $\text{In}(\text{C}_5\text{H}_4\text{CMe}_3)$ consists of infinite zigzag chains. There are no interstrand $\text{In}\cdots\text{In}$ interactions. The compound crystallizes in the monoclinic space group $\text{P2}_1/\text{n}$ with four formula units in the unit cell with dimensions of a 6.139(1)Å, b 9.765(6)Å, c 15.543(3)Å, β 92.87(1) and ρ_{calcd} 1.68g/cm³.



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Compounds which have a group 13 element in a low oxidation state are an interesting but little studied class of compounds. The most common indium(I) derivatives incorporate a cyclopentadienyl substituent and include $\text{In}(\text{C}_5\text{H}_5)$,^{1,2,3} $\text{In}(\text{C}_5\text{H}_4\text{Me})$,³ $\text{In}(\text{C}_5\text{H}_4\text{SiMe}_3)$,⁴ $\text{In}(\text{C}_5\text{H}_4\text{GeMe}_3)$,⁴ $\text{In}(\text{C}_5\text{Me}_5)$ ^{5,6} and $\text{In}[\text{C}_5(\text{CH}_2\text{Ph})_5]$.⁷ It is noteworthy that the crystalline state of all of these compounds except $\text{In}(\text{C}_5\text{H}_4\text{GeMe}_3)$ have been studied and five different structures have been observed for the five compounds studied. Three compounds, $\text{In}(\text{C}_5\text{H}_5)$,³ $\text{In}(\text{C}_5\text{H}_4\text{Me})$ ³ and $\text{In}(\text{C}_5\text{H}_4\text{SiMe}_3)$ ⁴, exist in the solid state as zigzag chains but with different numbers of short interstrand $\text{In}\cdots\text{In}$ interactions. Each indium in $\text{In}(\text{C}_5\text{H}_5)$ is 3.986(1)Å away from two other indium atoms³ whereas $\text{In}(\text{C}_5\text{H}_4\text{Me})$ has an indium atom at 3.986(1)Å from only one other indium atom.³ The trimethylsilyl derivative $\text{In}(\text{C}_5\text{H}_4\text{SiMe}_3)$ has no interstrand interactions.⁴ Pentamethylcyclopentadienylindium(I)^{5,6} exists as a hexameric(octahedral) cluster with $\text{In}\cdots\text{In}$ interactions of 3.963(1) and 3.943(1)Å. The compound $\text{In}[\text{C}_5(\text{CH}_2\text{Ph})_5]$ ⁷ forms quasi-dimeric units with indium-indium distances of 3.631(2)Å, the shortest observed to date. In this paper we extend this series with synthesis and complete characterization of $\text{In}(\text{C}_5\text{H}_4\text{CMe}_3)$.

The compound $\text{In}(\text{C}_5\text{H}_4\text{CMe}_3)$ was prepared from InCl and $\text{Li}(\text{C}_5\text{H}_4\text{CMe}_3)$ in diethyl ether by using the method of Peppe, Tuck and Victoriano² and was fully characterized by its physical properties, partial elemental analyses (C,H), reaction with dilute aqueous HCl, IR, ¹H NMR and mass spectroscopy and a single crystal X-ray structural study. The synthesis of the compound according to the following equation was observed to be typical of indium(I)



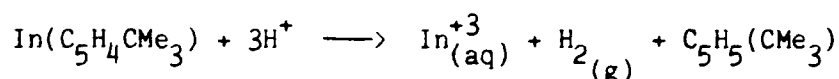
compounds. Upon addition of $\text{Li}(\text{C}_5\text{H}_4\text{CMe}_3)$, a gray colored material, possibly indium metal, was observed. The lithium cyclopentadienide reagent apparently acts as a reducing agent or the indium(I) disproportionates to a slight extent.⁸ However, the extent of decomposition was small. The colorless, crystalline indium(I) product was isolated in ~90% yield.

The structure of $\text{In}(\text{C}_5\text{H}_4\text{CMe}_3)$ consists of (theoretically) infinite zigzag chains based on $[\text{In}(\text{C}_5\text{H}_4\text{CMe}_3)]_\infty$ with no interstrand $\text{In}\cdots\text{In}$ interactions. The structure is very similar to that observed for $[\text{In}(\text{C}_5\text{H}_4\text{SiMe}_3)]_\infty$. The labelling of atoms in the basic $\text{In}(\text{C}_5\text{H}_4\text{CMe}_3)$ unit is illustrated in Figure 1 whereas the packing diagram is shown in Figure 2. Bond distances and bond angles are collected in Table 1. In the solid state each indium atom interacts with two (t-butyl)cyclopentadienyl ligands ($\angle \text{centroid}\cdots\text{In}\cdots\text{centroid} = 128.2$) and each cyclopentadienyl ligand is "inversely sandwiched" between two indium atoms ($\angle \text{In}\cdots\text{centroid}\cdots\text{In} = 173.4^\circ$). The corresponding angles in $\text{In}(\text{C}_5\text{H}_4\text{SiMe}_3)$,⁴ $\text{In}(\text{C}_5\text{H}_4\text{Me})^3$ and $\text{In}(\text{C}_5\text{H}_5)^3$ are 131.78 and 175.94° , 130.66 and 179.74° , and 128.02 and 176.99° , respectively. Thus, these intrachain angles are very similar for all compounds with zigzag chains (see Table II).

The basic carbocyclic ring, defined by atoms C(1) through C(5), is associated with In-C distances of $2.71(1)$ - $2.911(9)\text{\AA}$ (average 2.81\AA) and an $\text{In}\cdots\text{centroid}$ distance of 2.53\AA . These distances for $\text{In}(\text{C}_5\text{H}_4\text{CMe}_3)$ are shorter than for $\text{In}(\text{C}_5\text{H}_4\text{SiMe}_3)$ ⁴ of In-C(average) of 2.872\AA and $\text{In}\cdots\text{centroid}$ of 2.609\AA . The second carbocyclic ring (atoms $\text{C}(1)^* - \text{C}(5)^*$) is associated with In-C^{*} distances of $2.98(1)$ to $3.310(9)\text{\AA}$ (average 3.14\AA) and an $\text{In}\cdots\text{centroid}^*$ distance of 2.85\AA . The corresponding distances in

$\text{In}(\text{C}_5\text{H}_4\text{SiMe}_3)^4$ are shorter. Thus, $\text{In}(\text{C}_5\text{H}_4\text{CMe}_3)$ has the shortest In-C (In-centroid) and longest In-C* (In-centroid*) for any cyclopentadienylindium(I) derivative (Table II) with a zigzag structure.

The existence of indium in the + 1 oxidation state for $\text{In}(\text{C}_5\text{H}_4\text{CMe}_3)$ has been confirmed by its reactions with dilute aqueous HCl. The ratio of mol of H_2 /mol of In(I) compound was 0.957. These results are consistent with the following balanced chemical equation. However, it must be noted that



indium metal was found upon exposure of $\text{In}(\text{C}_5\text{H}_4\text{CMe}_3)$ to the acid. Then, the metal appeared to react with the acid to liberate H_2 . Similar observations have been noted for other cyclopentadienylindium(I) compounds.³⁻⁶

The mass spectrum of $\text{In}(\text{C}_5\text{H}_4\text{CMe}_3)$, which readily sublimates at 35° C, suggests the presence of a monomeric species in the gas phase as has been observed by electron diffraction studies for $\text{In}(\text{C}_5\text{H}_5)$,^{3,12} $\text{In}(\text{C}_5\text{H}_4\text{Me})^3$ and $\text{In}(\text{C}_5\text{Me}_5)^5$. The primary ions detected in the mass spectrum were $\text{In}^+(115\text{M/z})$ and $\text{In}(\text{C}_5\text{H}_4\text{CMe}_3)^+$ (236 M/z). Thus, one of the indium-cyclopentadienyl ring interactions of the solid must be quite weak.

Experimental Section

All compounds described in this investigation were exceedingly sensitive to oxygen and water and were manipulated in a standard vacuum line or under a purified argon atmosphere by using Vacuum/Atmospheres Dri-Lab. The cyclopentadienylindium(I) derivative was so exceedingly sensitive to trace quantities of moisture that all glassware used for their preparation, characterization, and handling was flame-heated under dynamic vacuum and then permitted to stand in the drybox for approximately 2h prior to use.

All solvents were purified by standard techniques. Indium(I) chloride was purchased from Strem Chemicals, Inc. and was used without further purification. The reagent $\text{Li}(\text{C}_5\text{H}_4\text{CMe}_3)$ was prepared from 2,6-dimethylfulvene and LiMe in OEt_2 as previously described.⁹ Elemental analyses were performed by Schwarzkopf Microanalytical Laboratory, Woodside, NY. Infrared spectra of Nujol mulls were recorded with a Perkin-Elmer 683 spectrometer. Absorption intensities are reported with the abbreviations w(weak), m(medium), s(strong), sh(shoulder), br(broad), and v(very). The ^1H NMR spectra were recorded immediately after sample preparation at 300 MHz with a Varian Gemini-300 NMR spectrometer. Chemical shifts are reported in δ units (ppm) and are referenced to SiMe_4 at δ 0.00 and benzene at δ 7.13. All NMR tubes were sealed under vacuum. Mass spectra of the samples contained in capillary tubes were recorded with a VG Model 70-SE spectrometer equipped with Electron Impact (EI) capabilities.

Synthesis of $\text{In}(\text{C}_5\text{H}_4\text{CMe}_3)_3$. A side-arm dumper charged with 0.712g (4.74 mmol) of finely ground InCl was connected to a two-neck flask containing 0.602 g (4.70 mmol) of lithium t-butylcyclopentadienide. Twenty-five mL of dry diethyl ether was distilled into the flask. While the solution was at room temperature, the InCl powder was added all at once to the rapidly stirred mixture. Some of the InCl decomposed to form a grey colored material, possibly indium metal. The mixture was then stirred for 19 h in the dark. The ether was removed very quickly by vacuum distillation. (No hot water bath was used to assist the distillation). An 80 ° elbow equipped with a Schlenk flask was attached to the original two-neck reaction flask in the dry box. The clean flask was placed in a -196°C bath and the product was dynamically sublimed from the reaction flask into the clean Schlenk flask. This isolation procedure took only 1 hour because of the volatility

of t-butylcyclopentadienylindium(I). The product was then transferred to a straight 20 mm Solv-SealTM tube. The tube was evacuated and was placed in a 35 °C oil bath for 6 h. The product sublimed under static vacuum as clear colorless crystals. The compound t-butylcyclopentadienylindium(I), $\text{In}(\text{C}_5\text{H}_4\text{CMe}_3)$ (1.006 g, 4.26 mmol), was isolated in 90.6% yield as based on $\text{Li}(\text{C}_5\text{H}_4\text{CMe}_3)$. $\text{In}(\text{C}_5\text{H}_4\text{CMe}_3)$: Colorless crystalline solid; mp 44.5 °C (glass transition), 47.5 - 48.5 °C (melting), ~300 °C (decomposition) pale yellow/brown. ^1H NMR (C_6D_6 , δ): 1.18 (s, 9H, CMe_3), 5.79 (t, 2H, CH, $^3J_{\text{HCCH}} = 2.8$ Hz), 5.96 (t, 2H, CH, $^3J_{\text{HCCH}} = 2.9$ Hz). IR (Nujol mull, cm^{-1}): 3091 (m), 1672 (vw, br), 1608 (w, br), 1562 (w, br), 1475 (s, sh), 1400 (m), 1270 (s), 1195 (m), 1181 (m), 1145 (vs), 1040 (m), 1030 (m), 1014 (m), 909 (m), 845 (w), 845 (w), 814 (vs, br), 754 (vs, br), 665 (vs), 575 (vw), 460 (w), 442 (s), 350 (m). Anal. Calcd: C, 45.79; H, 5.55. Found: C, 45.49; H, 5.49. MS EI 10V (M/z, relative intensity): 115, 100%, In^+ ; 236, 40%, $\text{In}(\text{C}_5\text{H}_4\text{CMe}_3)^+$.

Hydrolysis of $\text{In}(\text{C}_5\text{H}_4\text{CMe}_3)$. A quantity of $\text{In}(\text{C}_5\text{H}_4\text{CMe}_3)$ (0.104g, 0.440 mmol), was placed in a 100 mL round bottom flask. After this flask was evacuated while at -196 °C, ~20 mL of 3M HCl was added through an addition tube. Immediately upon exposure to aqueous acid, the compound decomposed to indium metal. Then, the metal apparently reacted with the acid. The hydrolysis was allowed to proceed for 3 days at ~100 °C. The noncondensable gas was isolated and measured by means of a Töepler pump - gas burette assembly. The volume of H_2 collected, 8.8 cmHg at STP, (0.421 mmol) corresponds to a 95.7% yield as based on the oxidation of indium(I) to indium(III) with formation of one mole of H_2 per mole of indium(I).

X-ray Data Collection, Structure Determination and Refinement for $\text{In}(\text{C}_5\text{H}_4\text{CMe}_3)$. A transparent single crystal was mounted in a thin-walled glass

capillary and transferred to the goniometer. The space group was determined to be the centric $P2_1/n$ from the systematic absences. A summary of data collection parameters is given in Table III.

The poor quality of the crystalline material available for study resulted in high esd's and a very poor goodness of fit parameter. The geometrically constrained hydrogen atoms were placed in calculated positions 0.95\AA from the bonded carbon atom and allowed to ride on that atom with B fixed at 5.5\AA^2 . The methyl hydrogen atoms were included as a rigid group with rotational freedom at the bonded carbon atom ($C-H = 0.95\text{\AA}$, $B = 5.5\text{\AA}^2$). Refinement of nonhydrogen atoms with anisotropic temperature factors led to the final values of $R = 0.071$ and $R_w = 0.072$. The final values of the positional parameters are given in Table IV.

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Table 1. Bond Distances (Å) and Angles (deg) for $\text{In}(\text{C}_5\text{H}_4\text{CMe}_3)$

Atoms	Distance	Atoms	Distance
In -- C(1)	2.837(8)	In -- C(2)	2.738(9)
In -- C(3)	2.71(1)	In -- C(4)	2.84(1)
In -- C(5)	2.911(9)	In -- C(1)*	3.207(9)
In -- C(2)*	3.310(9)	In -- C(3)*	3.20(1)
In -- C(4)*	2.98(1)	In -- C(5)*	2.98(1)
C(1) -- C(2)	1.42(1)	C(1) -- C(5)	1.43(1)
C(1) -- C(6)	1.51(1)	C(2) -- C(3)	1.40(1)
C(3) -- C(4)	1.42(1)	C(4) -- C(5)	1.41(1)
C(6) -- C(7)	1.52(1)	C(6) -- C(8)	1.51(1)
C(6) -- C(9)	1.53(1)	Cent -- In	2.53
Cent* -- In	2.85		
Atoms	Angle	Atoms	Angle
C(2) -- C(1) -- C(5)	107.5(8)	C(2) -- C(1) -- C(6)	125.7(8)
C(5) -- C(1) -- C(6)	126.6(8)	C(1) -- C(2) -- C(3)	108.2(9)
C(2) -- C(3) -- C(4)	108.4(9)	C(3) -- C(4) -- C(5)	107.8(9)
C(1) -- C(5) -- C(4)	108.1(9)	C(1) -- C(6) -- C(7)	111.6(8)
C(1) -- C(6) -- C(8)	109.7(8)	C(7) -- C(6) -- C(8)	110(1)
C(1) -- C(6) -- C(9)	110.0(8)	C(7) -- C(6) -- C(9)	107.6(9)
C(8) -- C(6) -- C(9)	108(1)	Cent -- In -- Cent*	128.2
In -- Cent -- In*	173.4		

Table II. Comparisons of Structural Features of Cyclopentadienylindium(I)
Compounds

	$\text{In}(\text{C}_5\text{H}_5)^{\text{a}}$	$\text{In}(\text{C}_5\text{H}_4\text{Me})^{\text{a}}$	$\text{In}(\text{C}_5\text{H}_4\text{SiMe}_3)^{\text{b}}$	$\text{In}(\text{C}_5\text{H}_4\text{CMe}_3)$
In-C (av)(Å)	2.959	2.867	2.872	2.81
In-C [*] (av)(Å)	2.924	3.016	3.067	3.14
In-centroid(Å)	2.726	2.609	2.609	2.53
In-centroid [*] (Å)	2.687	2.771	2.822	2.85
centroid-In-centroid(°)	128.02	130.66	131.78	128.2
In-centroid-In [*] (°)	176.99	179.74	175.94	173.4
mp (°C)	169.3-171.0	48.5-51.0	51.4-51.8	47.5-48.5
mmol H ₂ /mmol In(I)	0.942	0.955	0.867	0.956

a) Reference 3.

b) Reference 4.

Table III. Crystal Data and Summary of Intensity Data Collection and Structure Refinement.

Compound	In(C ₅ H ₄ CMe ₃)
Color/Shape	colorless/parallelepiped
Mol. Wt.	236.02
Space group	P2 ₁ /n
Temp., °C	20
Cell Constants ^a	
a, Å	6.139(1)
b, Å	9.765(6)
c, Å	15.543(3)
β, deg	92.87 (1)
Cell vol. (Å ³)	930.6
Molecules/unit cell (Z)	4
ρ _{calc} , g cm ⁻³	1.68
μ _{calc} , cm ⁻¹	24.5
Diffraction/Scan	Enraf-Nonius CAD-4/ω-2θ
Range of relative transm. factors, %	81/100
Radiation, graphite monochromator	MoKα(λ=0.71073)
Max crystal dimensions (mm)	0.13 x 0.35 x 0.53
Scan width (°)	0.80 + 0.35tanθ
Std reflctns	200; 0, 10, 0; 0, 0, 12
Decay of stds	± 2%
Reflctns measured	1939
2θ range (°)	2 ≤ 2θ ≤ 50
Range of h, k, l	+7, +11, ±18
Obsd reflctns	1370
Computer programs ^c	SHELX ¹⁰
Structure solution	Heavy atom techniques
No. of parameters varied	100
Weights	[σ(F _o) ² + 0.00001 F _o ²] ⁻¹
GOF	15.5
R = Σ F _o - F _c /Σ F _o	0.071
R _w	0.072
Largest feature final diff. map, eÅ ⁻³	1.0 (near In)

^aLeast-squares refinement of ((sinθ)/λ)² values for 25 reflections θ > 20°.

^bCorrections: Lorentz-polarization and absorption (empirical, psi scan).

^cNeutral scattering factors and anomalous dispersion corrections from ref. 11

Table IV. Final Fractional Coordinates for $\text{In}(\text{C}_5\text{H}_4\text{CMe}_3)$

Atom	x/a	y/b	z/c	B(eqv)a
In	0.9420(1)	0.49343(8)	0.75940(5)	3.58
C(1)	0.789(1)	0.2599(9)	0.6661(6)	1.96
C(2)	0.608(2)	0.3251(9)	0.7025(6)	2.44
C(3)	0.628(2)	0.308(1)	0.7921(7)	3.41
C(4)	0.817(2)	0.228(1)	0.8125(7)	3.15
C(5)	0.916(2)	0.198(1)	0.7349(6)	2.67
C(6)	0.828(2)	0.250(1)	0.5710(6)	2.42
C(7)	0.715(2)	0.126(1)	0.5302(7)	4.45
C(8)	1.070(2)	0.241(2)	0.5583(8)	4.89
C(9)	0.739(3)	0.377(1)	0.5240(7)	4.90

$$^aB(\text{eqv}) = 4/3[a^2B_{11} + b^2B_{22} + c^2B_{33} + ab(\cos\gamma)B_{12} + ac(\cos\beta)B_{13} + bc(\cos\alpha)B_{23}].$$

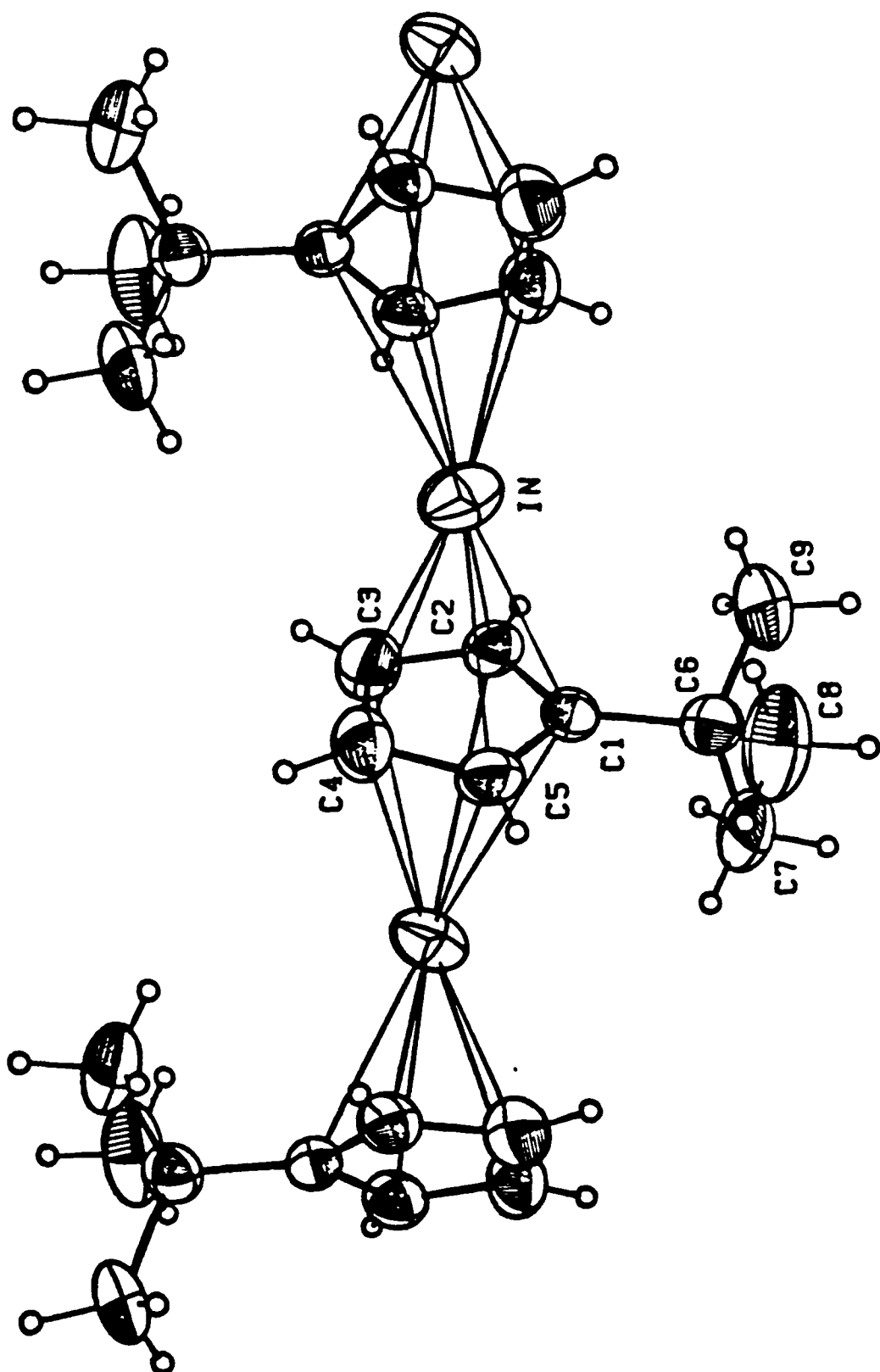
Captions to Figures

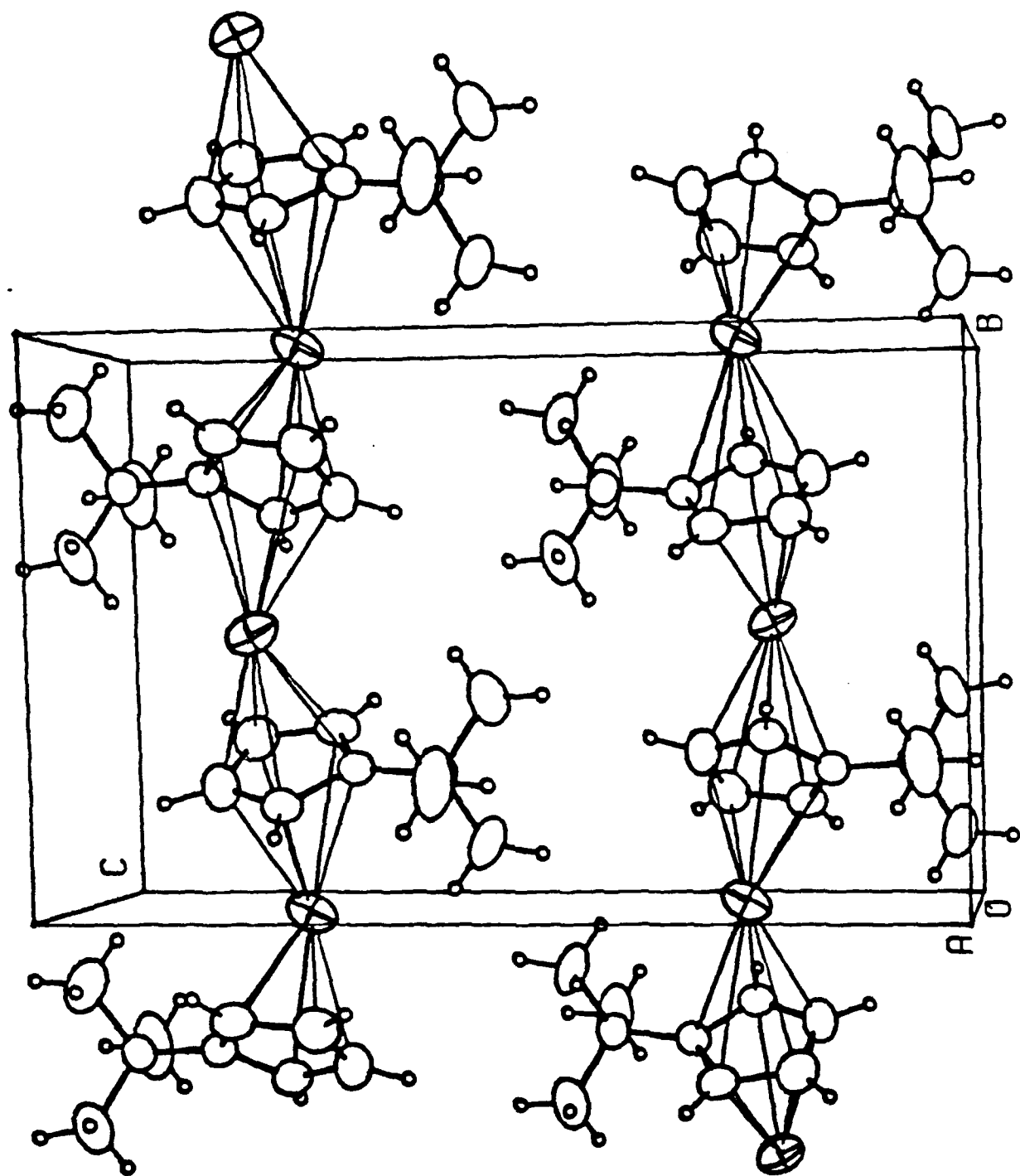
Figure 1 Labeling of atoms in $\text{In}(\text{C}_5\text{H}_4\text{CMe}_3)$ (ORTEP diagram; 50% ellipsoids with hydrogen atoms artificially reduced).

Figure 2 Packing diagram for $\text{In}(\text{C}_5\text{H}_4\text{CMe}_3)$ showing the infinite zigzag chains.

Table . Calculated Coordinates for Hydrogen Atoms in $\text{In}(\text{C}_5\text{H}_4\text{CMe}_3)$

Atom	x/a	y/b	z/c
H(1)[C(2)]	0.492	0.371	0.672
H(1)[C(3)]	0.530	0.343	0.832
H(1)[C(4)]	0.868	0.202	0.869
H(1)[C(5)]	1.047	0.148	0.730
H(1)[C(7)]	0.717	0.107	0.470
H(2)[C(7)]	0.569	0.145	0.545
H(3)[C(7)]	0.768	0.049	0.562
H(1)[C(8)]	1.125	0.161	0.586
H(2)[C(8)]	1.136	0.320	0.584
H(3)[C(8)]	1.099	0.238	0.499
H(1)[C(9)]	0.587	0.377	0.533
H(2)[C(9)]	0.762	0.383	0.464
H(3)[C(9)]	0.804	0.454	0.553





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